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NON-DESTRUCTIVE EVALUATION OF DEFECTS IN STRUCTURAL MATERIALS U--ETC(U)
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First Technical Report

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NON-DESTRUCTIVE EVALUATION OF DEFECTS IN STRUCTURAL MATERIALS USING LONG-WAVELENGTH NEUTRONS

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Small-angle scattering profiles from HSLA steels (Fe-0.05C-1.0Mn-0.12Nb-0.06Al) annealed at various temperatures ranging between 660°C and 1000°C were obtained. The values obtained from Guinier analysis and size distribution calculations were compared with those obtained from electron micrographic observations, and were shown to agree satisfactorily. Similar data were obtained from samples of Ni-P amorphous glasses annealed at 295°, 400°, and 500°C. No crystallization effects were observed from 295°C sample.

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The Guinier plots of the 400° and 500°C samples indicate good agreement with the expected behavior due to annealing of the amorphous state; namely, the higher annealing temperatures correspond to larger microcrystallite sizes.

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Abstract

Small-angle scattering profiles from HSLA steels (Fe-0.05C-1.0Mn-0.12Nb-0.06Al) annealed at various temperatures ranging between 660°C and 1000°C were obtained. The valued obtained from Guinier analysis and size distribution calculations were compared with those obtained from electron micrographic observations, and were shown to agree satisfactorily. Similar data were obtained from samples of Ni-P amorphous glasses annealed at 295°, 400°, and 500°C. No crystallization effects were observed from 295°C sample. The Guinier plots of the 400° and 500°C samples indicate good agreement with the expected behavior due to annealing of the amorphous state; namely, the higher annealing temperatures correspond to larger microcrystallite sizes.

Introduction

The non-destructive test methods currently used for the detection of structural flaws and defects are totally inadequate in identifying defect sizes below 1 μm in diameter. The objective of this research program is to develop a new non-destructive test method (small-angle neutron scattering, SANS) and to establish its application to defect and flaw detection in structural materials using long-wavelength neutrons.

Several well-known NDE techniques such as ultrasonic testing, radiography, magnetic particle, eddy current, microwave scanning, and liquid penetrant methods have been available to the engineering and manufacturing services. These techniques have been employed to detect the presence of defects in alloys as well as to obtain an estimate for their sizes. The minimum detectable defect size is about 3 to 5 μm for the liquid penetrant technique, suitable for surface and near-surface flaws, and increases to a value of nearly 1 mm for radiography and ultrasonic methods, which are sensitive to defects in the bulk of the material. It is invariably difficult to metallurgically rejuvenate a structural component when a flaw is in size ranging to several micrometers. The sensitive methods of determining defects by the measurement of ultrasonic attenuation or radiography can only be utilized for rejection or acceptance of structural components. These characteristics of the present NDE techniques pose serious limitations on their application to high-cost components, such as turbine blades and disks, wing spars and bulk heads, which could otherwise be saved from premature failure through appropriate thermomechanical treatment to increase their service life.

From the point of view of cost effectiveness, it is necessary to develop a NDE technique capable of distinquishing defects in structural materials in their subcritical stages of formation. Not only would this be of significant value in identifying small defects, but also as a means of detecting critical changes in material characteristics well in advance of failure, thus safeguarding against possible costly damage to major structures and components.

Scattering behavior of long wavelength neutrons by solids at low angles (SANS) has been known for at least two decades (1,2). Its application to polymer physics and biological sciences has been well-known (3-5). SANS has only recently been applied to the study of defects in metals and alloys at Institut Max v. Lave-Paul Langevin, Grenoble, France, and Institut für Festkörperforschung, Kerforschungsanlage, Jülich, W. Germany has illustrated the potential use of SANS in evaluating various types of processing and in-service initiated defects in engineering alloys. All such studies, however, have been limited in scope due to the lack of sufficient reactor time for long wavelength neutron beam as well as the absence of a systematic research activity directed to the evaluation of defect size and distribution in solids.

Experimental

The NBS Reactor Facility (NBSR) is in the process of several major improvements in its SANS spectometer by incorporating state-of-the-art capabilities. These consist of a) replacement of the linear position-sensitive detector by an area detector, b) implementation of new focusing collimation geometry to enhance the

resolution without loss of intensity and c) addition of the cold source to the reactor to moderate the fast neutrons and to increase the thermal neutron flux. These improvements will effectively reduce the counting time by a factor of about 5 or 10. Based on collaboration and consultation with NRL scientists, the NBS-SANS spectometer will be designed in such a way as to accommodate samples in various sizes and configurations to allow, in a limited scale, NDE examination of small structural components without damaging them. The most significant of these design parameters is reflected in the improved resolution of the instrument (from $\Delta q = .010\ A^{-1}$ to $\Delta q = .003\ A^{-1}$) which, while of no major concern in polymer physics, plays an important part in materials defects program.

Because of a serious mechanical problem in the operation of the reactor, involving the control of the fuel rods, the NBS reactor has been inoperative since early August 1979. Tests on samples of superplastic zinc (Zn-22%Al) have therefore been delayed. Compounding the difficulty has been the fact no other research reactor in the U. S. has been ready for such measurements, nor will it be suitable for wide-range structural defects studies of interest to the materials program.

The NBS-SANS instrument is expected to restart by early March 1980 using the original system. Within 3 or 4 months thereafter, it is planned for a changeover to the new modified version. This changeover is expected to cause additional delays of a few months in the progress of the present work with superplastic alloys. Meanwhile, the data processing algorithms have

been developed and will be discussed in the results and discussion section of this report.

Results and Discussion

A) High Strength-Low Alloy Steels

In order to examine the capabilities of SANS in delineating various distribution of defects in materials, a study was undertaken to measure the effects of niobium carbonitride precipitates in a high-strength, low-alloy steel, (Fe-0.05C-1.0Mn-0.12Nb-0.06Al). Square samples 12 mm x 12 mm x 2.4 mm thick were prepared from rolled sheets of the material obtained from Inland Steels. The samples were chemically cleaned, and annealed in vacuum for 2 hours at various temperatures ranging between 600°C to 1000°C. By these annealing treatments, a variety of precipitate size and density distribution were obtained, ranging from a relatively small size, 100Å, and low densities to larger sizes, 300-500Å, and high densities, and subsequently to largest sizes, 600-1000Å, and low densities. Electron micrographs in Figure 1 reveal the NbCN precipitates in HSLA matrix. The electron microscopic evaluation of particle size and density was used to compare with the results non-destructively obtained from identical samples used for SANS measurements.

Practically all SANS data are monotonically decreasing functions of angle or "channel number." Figure 2 shows typical data obtained from one of the samples analyzed. Two aspects of SANS measurements are displayed in the figure. One is the order of magnitude of the intensities involved; the other is the effect of magnetic scattering resulting from the non-aligned domain structure of the ferromagnetic samples. The central portion of the

profile is an artifact due to the effect of the masks placed over the detector to eliminate the overload ("flooding"), which is caused by the presence of the direct beam in the spectrum. In actual data analysis, the two halves of the data are folded together and corrected for background and geometrical effects (beam divergence). The effect of the magnetic scattering on the SANS profile is quite significant and, depending on the degree of magnetism, may affect the data by an order of magnitude. Such an effect is certainly undesirable and must be identified in the study of microdefects in structural alloys. To eliminate the magnetic scattering the sample is magnetized by a saturating field in a direction perpendicular to the beam and parallel to the detector axis.

The data processing algorithm consists of the following routines:

a) "DECODE" in which the two halves of SANS profile are folded together, and corrected for background, which is measured separately. Each set of data is then corrected for transmission and absorption effects in order to normalize the values for comparison with other data in the same set.

b) "SMOOTH" In this program local and statistical fluctuations are eliminated, so that the general behavior of the data can be observed; the data is then ready for geometrical correction.

c) Beam divergence routines "DESMER" and "SLIT." These two programs introduce the correction for the horizontal (slit width) and vertical (slit length) divergences in the incoming beam.

d) "GUINIE" This program generates Guinier plots from the output of "SLIT" program and permits the examination of data for input in the size-distribution calculation.

The natural logarithm of the relative intensities from the samples annealed at five temperatures are plotted as a function of the square of the scattering angle θ (more precisely, $2\pi\theta/\lambda$) in the Guinier plot (6). This plot has the property that for sufficiently small angles, its slope represents the relative radius of gyration of the precipitates through the relationship

$$\text{slope} = -\frac{R^2}{3}.$$

For samples whose data have been normalized, the relative densities of various precipitate sizes can be compared on the Guinier plot observing relative scattered intensities at various points.

Several important features can be immediately discerned from the plots (Fig. 3). For example, the overall scattered intensity is higher for the 700°C sample than for any other sample. On the other hand, the intensities at very small angles (corresponding to very large particles) are higher for 1000°C sample than for 600°, 800° and 900°C. The major portion of all curves shows a relatively straight-line behavior corresponding to approximately 250-300Å. In general SANS results are in good agreement with the electron microscopic observations.

As an alternative to the Guinier plot, the scattering data has been analyzed using the particle size distribution calculations, in which the total scattering profile is considered to be the sum of intensities from a continuous distribution of particle sizes. This type of evaluation has been performed by other

investigators (7-9) using different algorithms. The result of such a calculation using a routine developed at NRL shows a comparison between samples annealed at 600°C and at 800°C, for the same range of precipitate sizes (Fig. 4). This method of evaluation, consistent with the Guinier analysis and electron microscopic data, gives the most comprehensive quantitative values of particle size and distribution from the bulk region of a solid.

B) Effect of Annealing on the Crystallization Behavior in Amorphous Ni-P Glasses

This investigation was originally performed at a single annealing temperature on a sample of Ni-P amorphous glass. The comparison between crystallized and amorphous states was shown both by x-ray diffraction and by small-angle neutron scattering. Following a modification of the collimation system of the SANS apparatus the data could be extended to much larger scattering angles resulting from increased counting time and improved statistics. A new set of measurements were performed in which the annealing temperature was varied on three samples at 295°C, 400°C and 500°C, respectively. The first sample (295°C) did not exhibit any SANS or XRD effects distinct from amorphous state. However, the SANS profiles of the 400°C and 500°C anneals were significantly different, although their XRD data did not reveal any discernible changes in crystallite size or other structural parameters. The extended data plotted on a Guinier plot for the 400°C and 500°C annealed samples are shown in Figure 5. As can be seen, the low angle intensities, corresponding to larger crystallite sizes, are higher

for the 500°C annealing temperature, compared to 400°C annealing. Conversely, the high angle data corresponding to smaller crysallites are higher for 400°C sample, compared to the 500°C sample. These differences are consistent with the expected growth of crystallites with annealing temperature. Note that the crystallite sizes (100-300Å) are especially well-suited for observation by SANS.

Future Plans

The SANS measurements of creep induced voids on superplastic alloys Z-200 (Zn-22Al) and Z-400 (Zn-22Al-0.5Cu-.01Mg) as a function of strain rate and temperature will commence immediately after the reactor facility at NBS is operational. In addition studies will be conducted by mid-FY80 to:

1) Establish the capabilities of the new SANS facility with area-sensitive detector at NBS as applied to materials defect program.

2) Analyze the effect of periodic precipitate distribution (Bragg scattering) on SANS profiles.

3) Evaluate porosities in hot consolidated powder alloys.

Additional Comments: Status of the NBS Reactor

Since early August 1979 up to the present time, the NBS reactor has been shut down due to a critical mechanical difficulty. The reactor is tentatively expected to restart by the end of February or early March 1980. The NBS SANS facility will operate in the old configuration (with the linear position sensitive detectors) for a period of 3-4 months, following which it will be converted to an intermediate configuration with a small

(250 mm x 250 mm) area position sensitive detector. This conversion is again expected to take a period of approximately 2 months. The final configuration will be available about Fall 1980, at which time the measurements are expected to proceed far more rapidly.

Acknowledgments

The size distribution calculations for HSLA steels (Fig. 4) were performed by J. Konnert and P. D'Antonio of Code 6030 (Laboratory for Structure of Matter), NRL, is acknowledged. Assistance of J. Reed of Code 6390, NRL, in electron microscopy and C. Vold, Code 6320, NRL, in the development of computer subroutines is greatly appreciated.

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Figure Captions

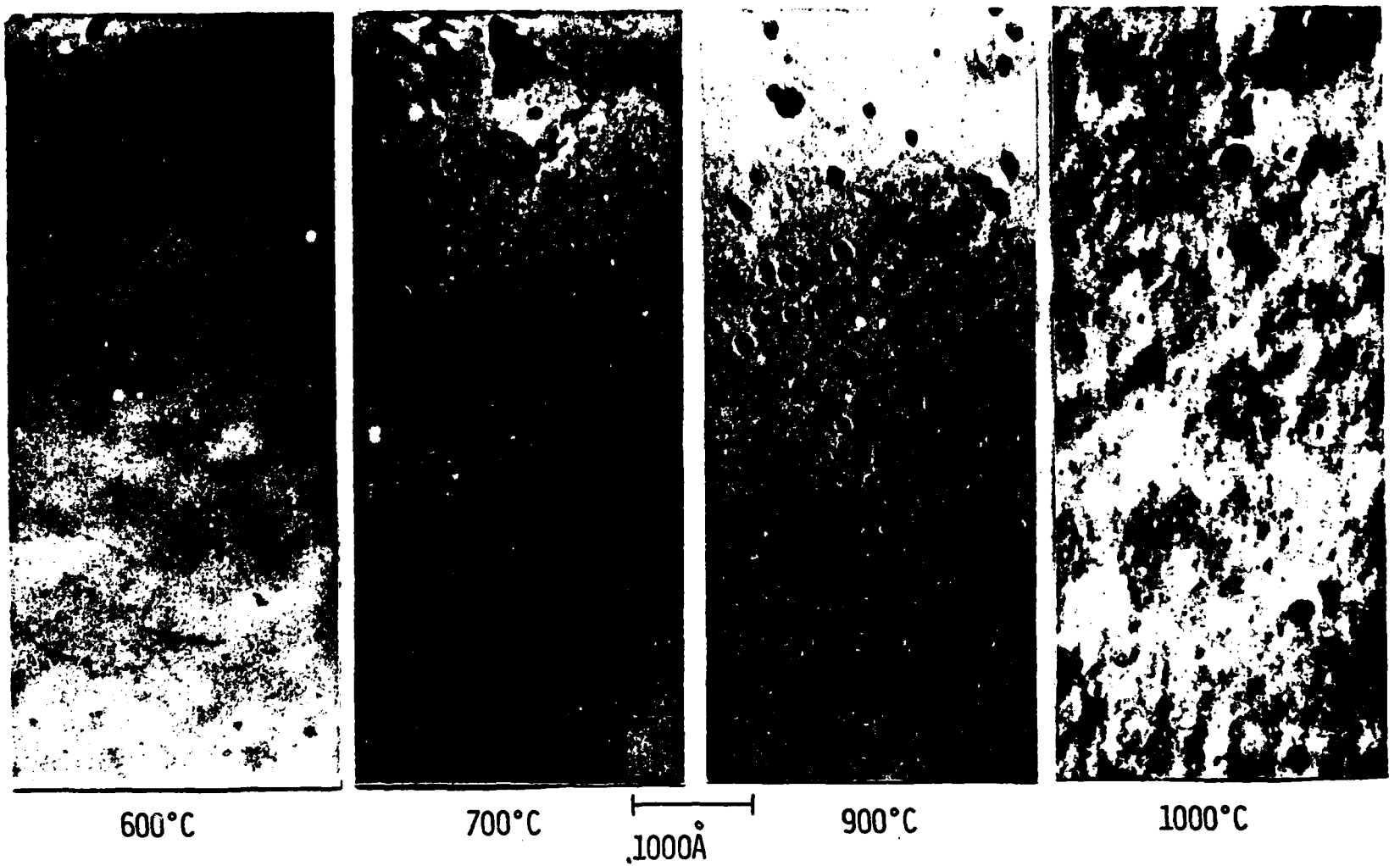
Figure 1 Temperature-dependent size distribution of Niobium carbonitride precipitates in an HSLA steel

Figure 2 Scattering profiles of an HSLA steel sample showing the order of magnitude of intensities and the effect of magnetic scattering

Figure 3 Guinier plots of SANS data from HSLA steels

Figure 4 Size distribution calculations from 600°C annealed and 800°C annealed HSLA samples

Figure 5 Guinier plots of SANS profiles from Ni-P amorphous glasses



TEMPERATURE DEPENDENT SIZE-DISTRIBUTION OF COLUMBIUM-CARBONITRIDE PRECIPITATES IN A HSLA STEEL

FIGURE 1 - TEMPERATURE-DEPENDENT SIZE DISTRIBUTION OF NIOBIUM CARBONITRIDE PRECIPITATES IN AN HSLA STEEL

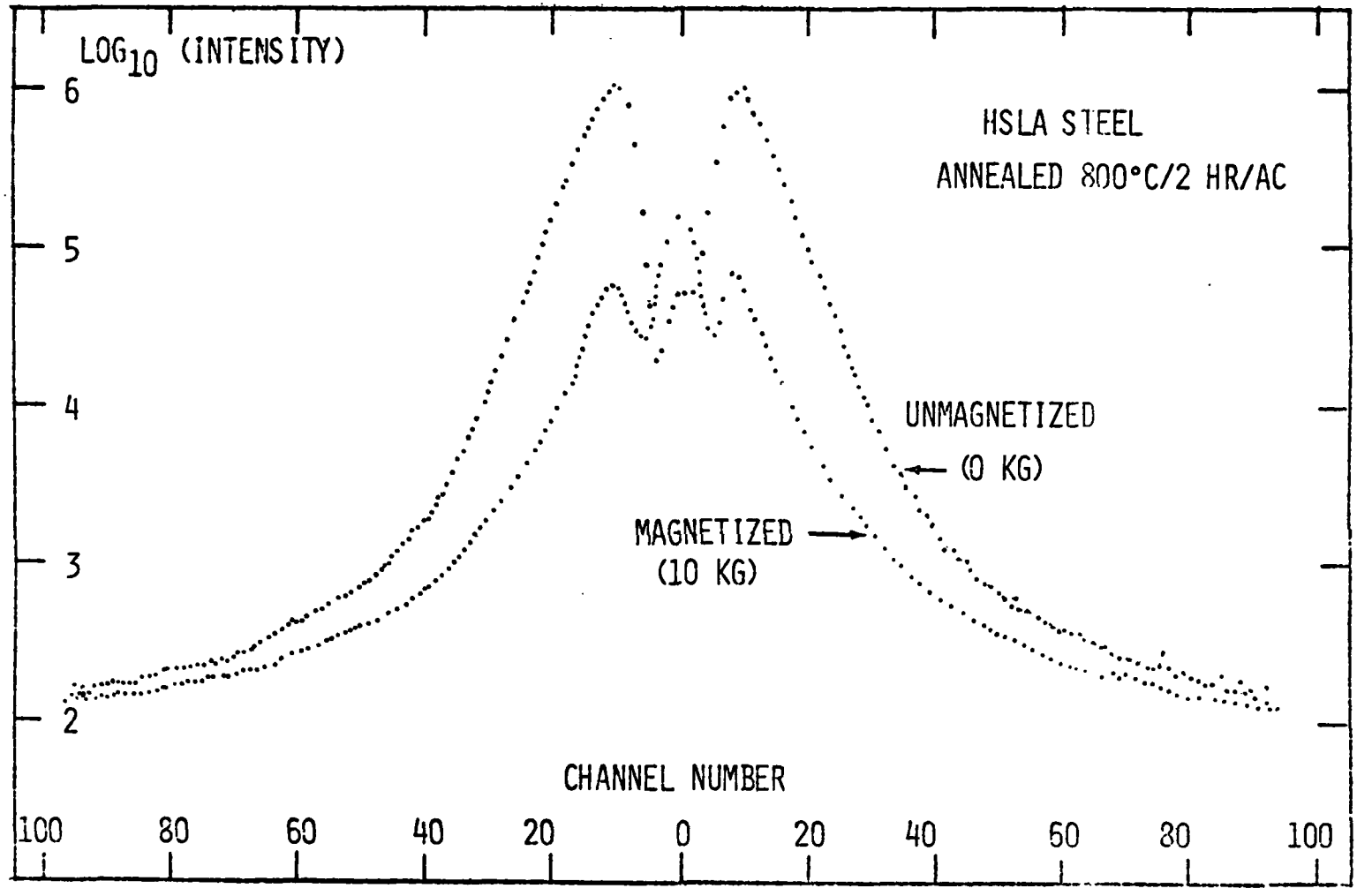


FIGURE 2 - SCATTERING PROFILES OF AN HSLA STEEL SAMPLE SHOWING THE ORDER OF MAGNITUDE OF INTENSITIES AND THE EFFECT OF MAGNETIC SCATTERING

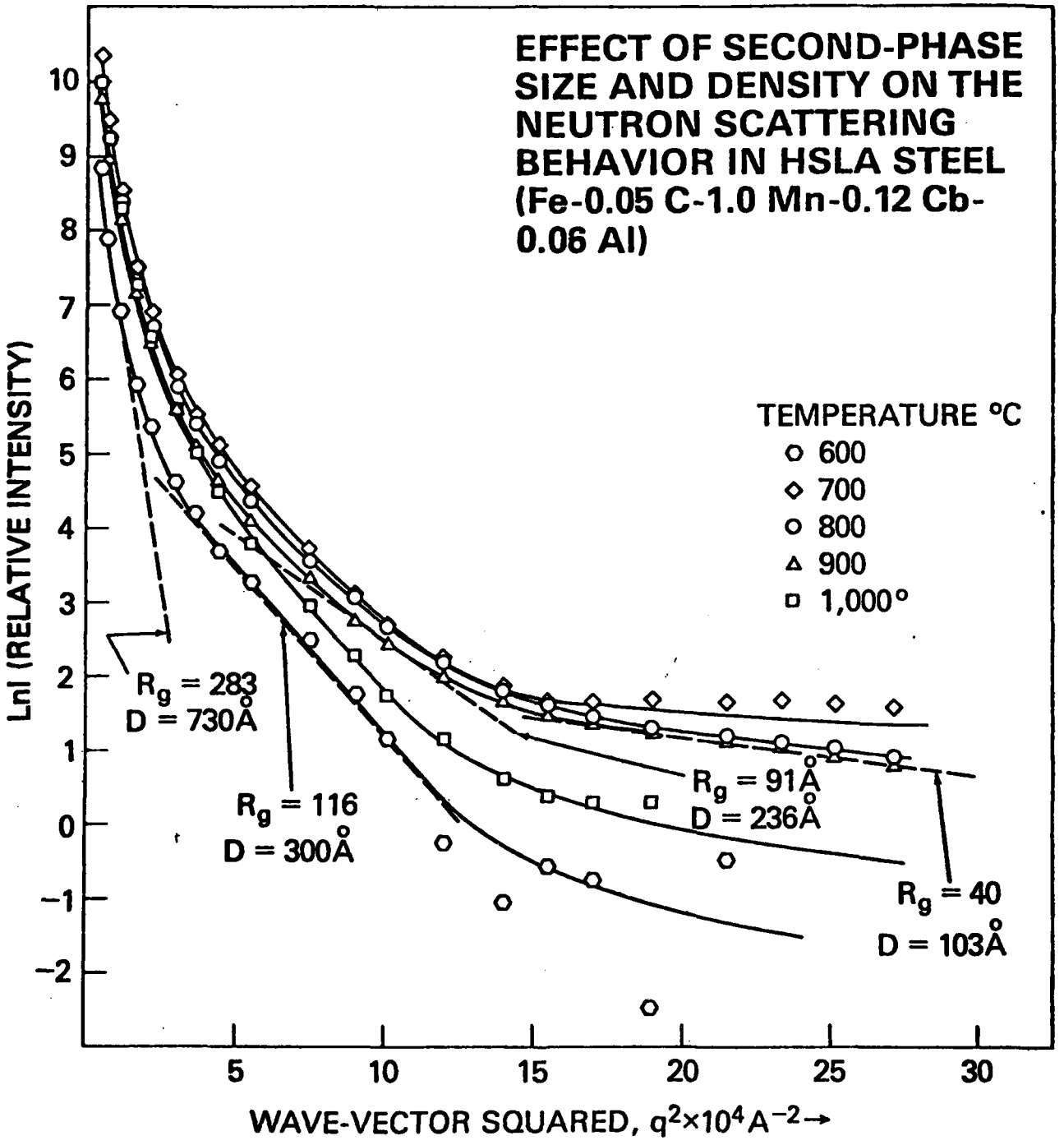


FIGURE 3 - GUINIER PLOTS OF SANS DATA FROM HSLA STEELS

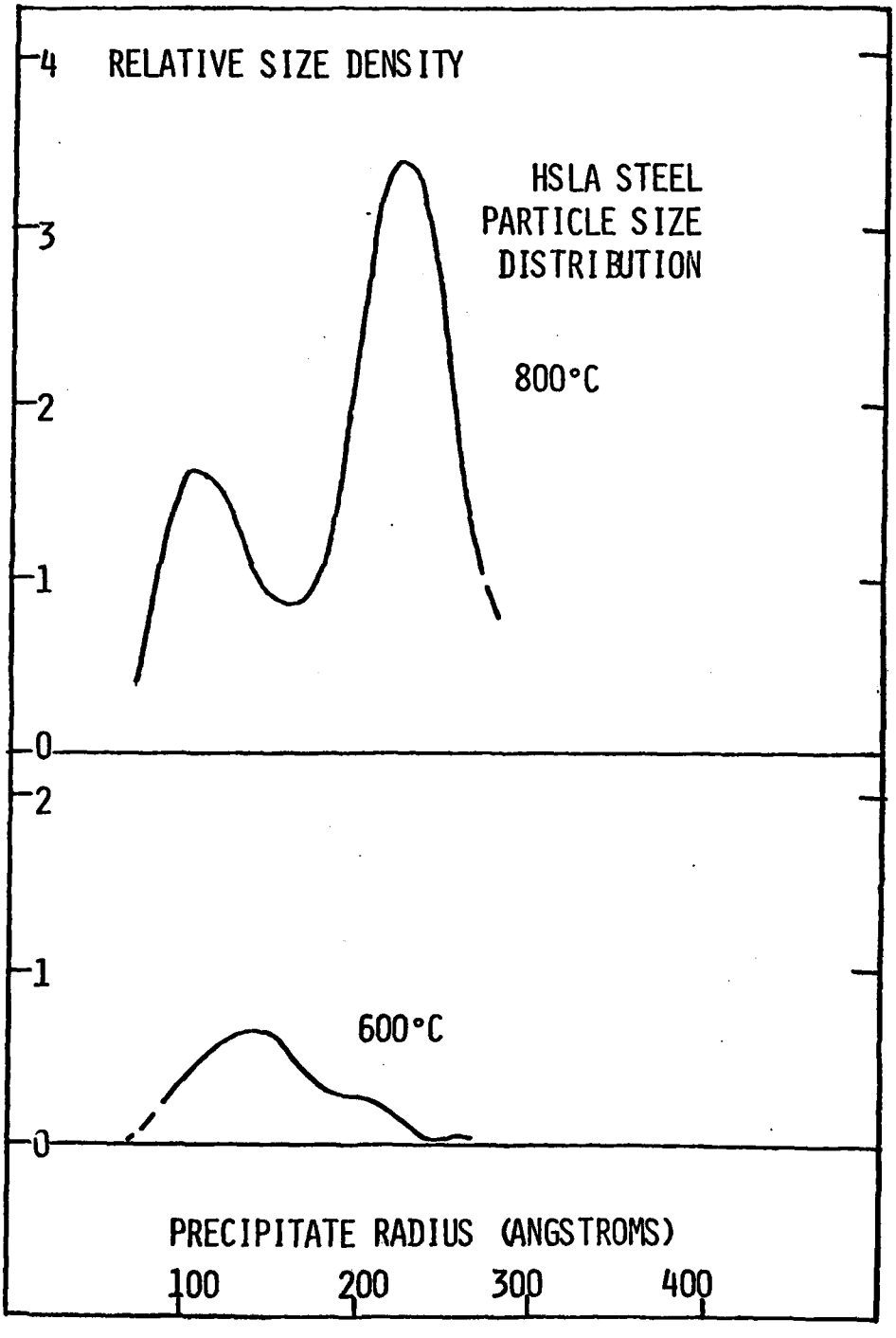


FIGURE 4 - SIZE DISTRIBUTION CALCULATIONS FROM 600°C ANNEALED AND 800°C ANNEALED HSLA SAMPLES

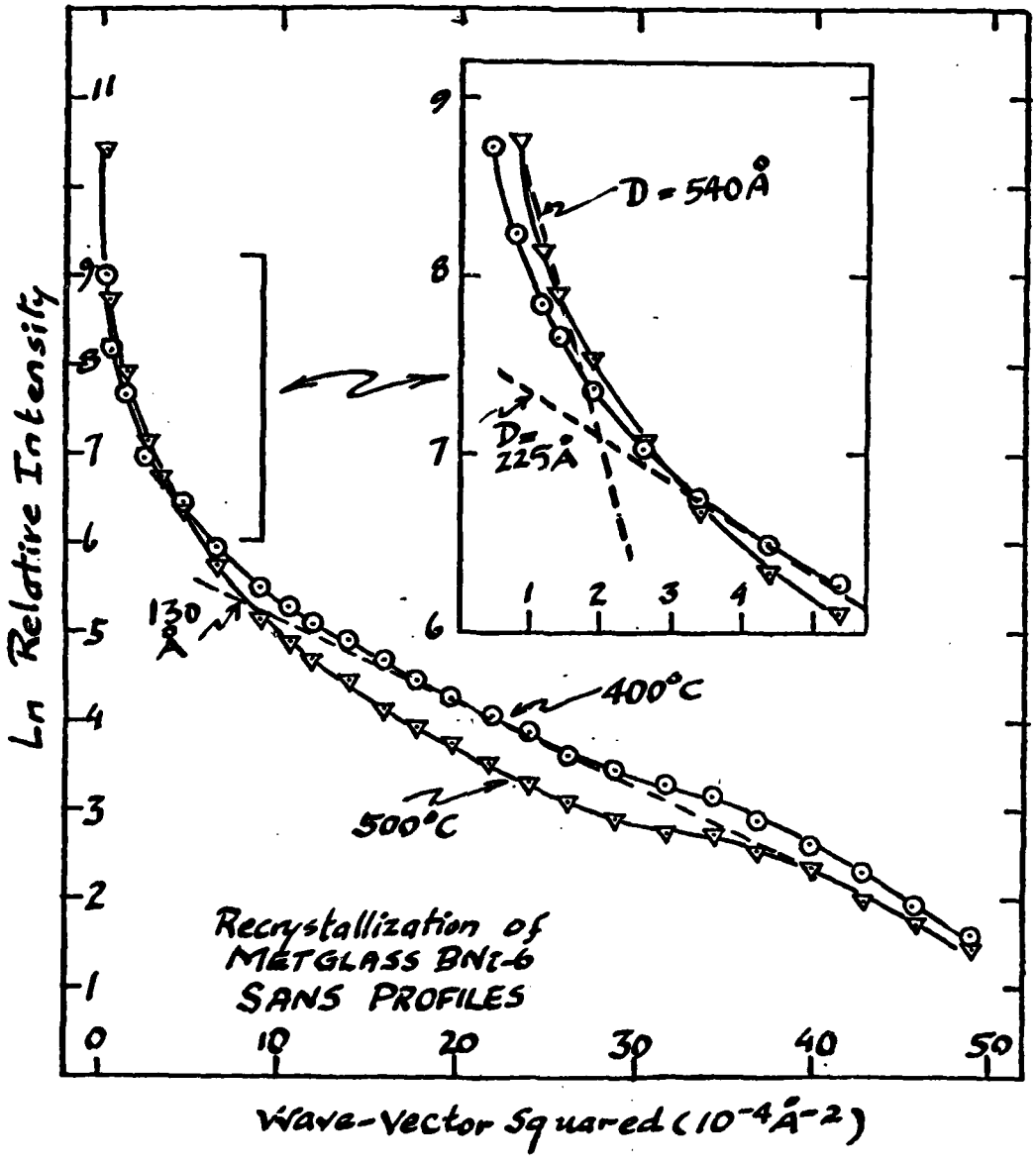


FIGURE 5 - GUINIER PLOTS OF SANS PROFILES FROM NI-P AMORPHOUS GLASSES